#### REPORT DOCUMENTATION PAGE

AFRL-SR-AR-TR-03-

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1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE	A. J. Same	3. DATES COVERED (From - To)
27-08-2003	Final Report	<b>1</b> 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	Feb. 15,2000-April 30, 2003
4. TITLE AND SUBTITLE			5a. CONTRACT NUMBER
DC Large Volume Non-Therma	l Plasma at Atmosph	neric Pressure	
			5b. GRANT NUMBER
			F49620-00-1-0168
			5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)			5d. PROJECT NUMBER
Dr. Mounir Laroussi			
			5e. TASK NUMBER
			2301/EX
			5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Old Dominion University			8. PERFORMING ORGANIZATION REPORT NUMBER
	ODURF		
	Hughes Hall, C	ld Domion Univ.	
	Norfolk, VA 2		
	<b></b>		
9. SPONSORING / MONITORING AGENCY	NAME(S) AND ADDRESS(ES	5)	10. SPONSOR/MONITOR'S ACRONYM(S)
Air Force Office	4015 Wilson B		AFOSR
Of Scientific Research	Rm. 713		
	Arlington, VA	., 22203-1954	11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION / AVAILABILITY STATEMENT

Unlimited

13. SUPPLEMENTARY NOTES

20030915 030

#### 14. ABSTRACT

This document is the final report on the research activities carried out under AFOSR Grant F49620-00-1-0168. It describes the research results and the various events, which took place during the period of this research program. The focus of our activities is the investigation of the properties of a large volume, non-thermal, atmospheric pressure plasma generated by the "Resistive Barrier Discharge". The biological applications of this discharge have also been investigated. These include the study of the germicidal effects as well as the effects of the plasma on the biochemical pathways of bacteria.

DISTRIBUTION STATEMENT A
Approved for Public Release
Distribution Unlimited

## 15. SUBJECT TERMS

Glow discharge, Atmospheric pressure, resistivity, current, DC voltage

16. SECURITY CLASSIFICATION OF:		17. LIMITATION	18. NUMBER	19a. NAME OF RESPONSIBLE PERSON	
		OF ABSTRACT	OF PAGES	Dr. Robert J. Barker	
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified	Unlimited		<b>19b. TELEPHONE NUMBER</b> (include area code) (703) 696-8574

#### Final Report

# DC Large Volume Non-Thermal Plasma at Atmospheric Pressure

# AFOSR Grant F49620-00-1-0168

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Period Covered: February 15, 2000 to April 31, 2003

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#### **Abstract**

This document describes the various activities carried out for and in support of the experimental research program, supported by AFOSR grant F49620-00-1-0168. The objective of this research program is to characterize a novel non-thermal discharge at atmospheric pressure (the Resistive Barrier Discharge, RBD), and to investigate its potential applications. Non-thermal plasmas at atmospheric pressure have direct applications in microwave communications, in shielding defense equipment against directed EM weapons, in biological and chemical warfare countermeasures, and in several industrial processes.

The Discharge under investigation produces large volume, steady-state plasmas between two electrodes covered by a special high resistivity material. The electrodes can be driven by a DC or AC (60 Hz) power source. Only a relatively moderate amount of input power (few hundred Watts) is necessary to maintain a plasma at atmospheric pressure with a number density in the  $10^{11}$ - $10^{12}$  cm<sup>-3</sup> range.

The research was carried out at The Applied Plasma Technology Laboratory of Old Dominion University. Collaboration with ODU's Department of Ocean, Earth, and Atmospheric Sciences was established to conduct work on the biological applications of the discharge.

#### **Research Activities**

During the duration of this grant a number of activities took place. First, a compact discharge reactor based on the "Resistive Barrier Discharge" (RBD) was built. The RBD is a novel discharge, developed by the PI and co-workers, with a configuration similar to the Dielectric Barrier Discharge (DBD). The added advantage of the RBD is the possibility to operate it in DC or quasi-DC (AC, 60 Hz). In this work, the RBD reactor was designed with the intentions that it will be used extensively in "biology-related" experiments. These experiments were a part of an ongoing study of the potential of non-thermal plasmas to interact with the cells of microorganisms. Figure 1 shows the general configuration of the RBD reactor.

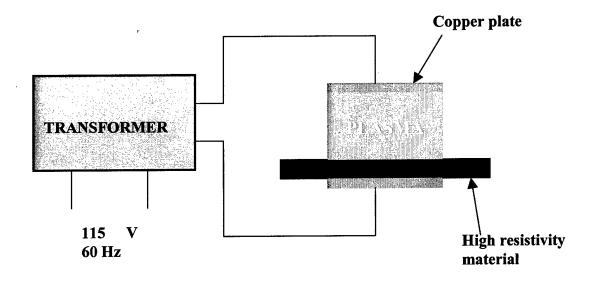


Fig. 1 General configuration of the RBD reactor

The plasma in the RBD reactor can be driven by either DC or AC (60 Hz) power source. Helium gas is used alone or as part of gas mixture in our experiments. Helium helps keep the discharge in the diffuse mode. Figure 2 shows a photograph of a discharge driven by an AC source and with a helium/air mixture.

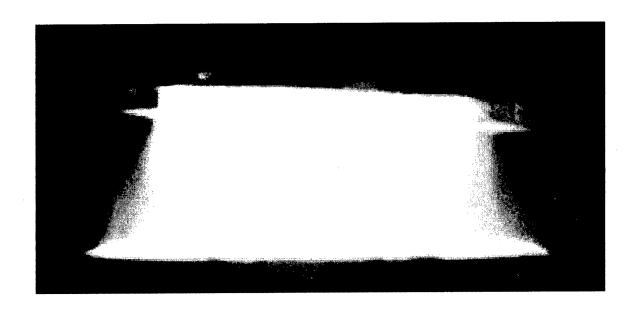


Fig. 2 Photo of an AC-driven RBD discharge in helium/air mixture

## **Discharge Characteristics**

#### I-V Characteristics and Light Emission

To characterize the RBD discharge we looked at its current and light emission. The current waveform revealed that even when the discharge was driven by a DC voltage, the current was pulsed. Our experiments showed that the current signal exhibited a pulsed form with pulses few microseconds wide at a repetition rate of few tens of kHz. This suggests that when the discharge current reached a threshold value, the voltage drop across the resistive layer became large to the point where the voltage across the gas became insufficient to ignite the discharge. Therefore, as the discharge extinguished, the current dropped rapidly and the voltage across the gas increased to a value capable of initiating the discharge again. To verify this we used a photomultiplier tube (PMT) to monitor the discharge light emission. Figure 3 shows both the discharge current and the PMT output signal. It is clear that there is correlation between the current pulses and the light pulses. In addition, it is interesting to note that the current pulses are not similar, especially in magnitude. This suggests that the discharge was not temporally uniform, and that both its number density and light emission fluctuated in time.

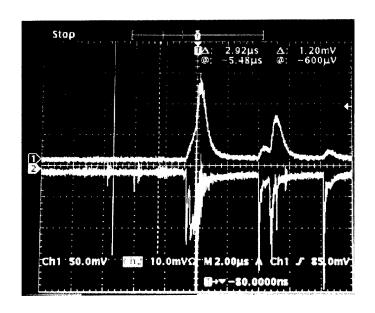


Fig. 3 Discharge current (yellow, top) and PMT signal

An archival paper describing the characteristics of the RBD was published in the IEEE Transactions on Plasma Science Special Issue on Images in Plasma Science (Vol. 30, No. 1, pp. 158-159, 2002). A copy of the manuscript is included in the Appendix of this report.

## Plasma Uniformity and Stability

The discharge could be run for more than half an hour at a time without apparent development of instabilities. However, if the resistive layer covering the electrodes undergoes localized heating (this usually occurs when helium is the smaller fraction of the helium/air mixture), it develops cracks, which could lead to arcing. Figure 4 shows an arc developing between the edges of the electrodes.

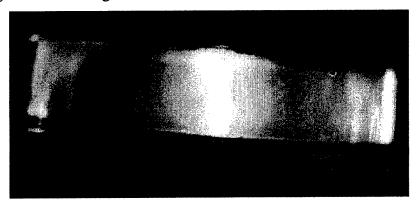


Fig. 4 Development of an arc at a localized defect in the resistive layer

As the amount of air (or oxygen) in a helium/oxygen (air) mixture increased, the spatial uniformity of the plasma started to deteriorate: random filaments formed at various locations across the electrodes. The filamentary structure of the discharge was accompanied with an increase in the electrode temperature. This situation can lead to localized defects in the resistive layer covering the electrodes (as mentioned earlier.) Figure 5 shows filaments randomly distributed within a rather diffuse plasma background. The gas mixture was 97%-3% helium/oxygen respectively.

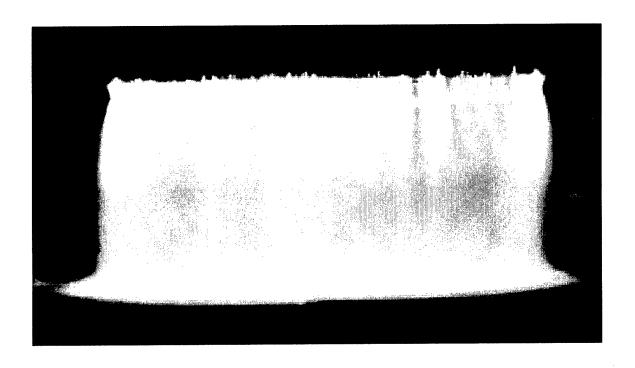


Fig. 5 Non-uniform plasma structure for a 97%-3% helium/oxygen mixture

## **Biological Applications**

In collaboration with ODU's Department of Ocean Earth and Atmospheric Sciences, experiments were conducted on the effects of the non-thermal plasma generated by the RBD on bacteria. These experiments were carried out by a Graduate Student (Mr. Paul Richardson). Mr. Richardson not only studied the germicidal effects of the plasma, but he also investigated the effects of the discharge on the biochemical pathways of bacteria and on their cell morphology. It was discovered that non-thermal plasmas exhibit sub-lethal effects, which could induce changes in enzyme activity. These changes can have direct impact on the metabolism of plasma-treated bacteria. Figure 6 and Figure 7 show the cases of increased and decreased heterotrophic activity, respectively. Also, using electron microscopy, it was discovered that the effects of plasma on cell morphology was dependent on the type of microorganism. Apparently, when exposed to a lethal plasma dose, Gram negative bacteria undergo substantial cell damage, while Gram positive bacteria show no visible morphological changes. Figure 8 and 9 show the case of Escherichia coli (Gram negative) and Bacillus subtilis (Gram positive) respectively. This work was presented at the IEEE Pulsed Power Plasma Conference in Las Vegas, Nevada (June, 2001), and at the International Symposium on Plasma Chemistry in Orleans, France (July, 2001). In addition, archival papers were published in the IEEE Transactions on Plasma Science (Special Issue on the Non-Thermal Medical/Biological Applications of Ionized Gases and Electromagnetic Fields, August, 2002) and in Applied Physics Letters (Vol. 81, No. 4, pp. 772-774, 2002).

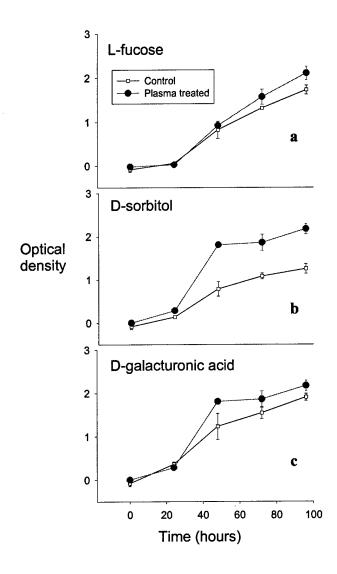


Fig. 6 Increased utilization of sole source carbon substrate by plasma treated E. coli cells. **a**, 1-fucose. **b**, d-sorbitol. **c**, d-galacturonic acid

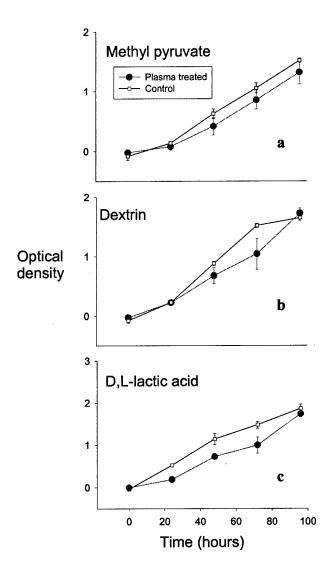
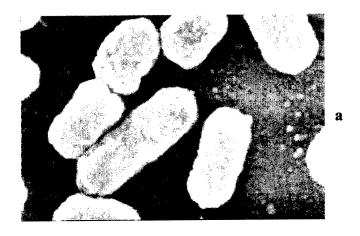


Fig. 7 Decreased utilization of sole source carbon substrates by plasma treated  $E.\ coli$  cells. **a**, methyl puryvate. **b**, dextrin. **c**, d,l-lactic acid.



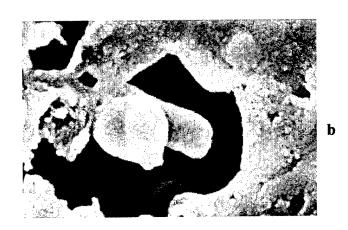


Fig. 8 Scanning Electron Microscope micrographs of *E. coli* cells. **a**, control. **b**, plasma treated.



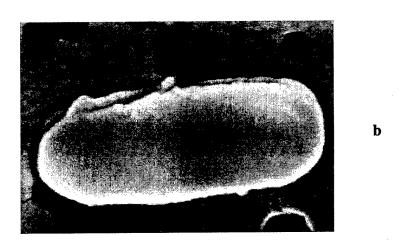


Fig. 9 Scanning Electron Microscope micrographs of *B. subtilis* cells. **a**, control. **b**, plasma treated

## **Development of a Novel Air Discharge**

It was always our intention to develop a method by which we can actually generate and maintain a non-thermal plasma in atmospheric pressure air at relatively low temperatures and low power requirements. In this context, we started investigating other means to achieve that goal. Recently we developed a new discharge configuration, which has showed some promise. The preliminary results obtained during the end period of this research grant indicated that with further work this method can go a long way in achieving our goal. Figure 10 shows the general configuration of the experimental setup.

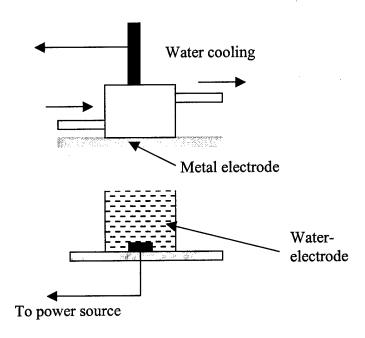


Fig. 10 Experimental setup

The air plasma is generated in a gap between a disk-shaped metal upper electrode and the surface of a water volume contained in a glass dish. An electrical connection to the power source is provided at the bottom of the water dish (see Fig. 10). The upper metallic electrode is water-cooled. When the plasma is switched on, evaporation and surface tension allows the creation of a thin layer of water attached to the surface of the upper electrode. Hence, the discharge becomes practically between two water surfaces. The discharge is ignited by applying a high AC (60 Hz) voltage (<20 kV) between the metal electrode and the water electrode. The air gap between the two electrodes is variable. Gaps up to 3 cm have been routinely used. The plasma does not fill the whole volume between the electrodes but rather assumes the shape of a cylindrical column and sometimes a conical column, 3 to 10 mm in diameter, depending on the magnitude of the

applied voltage. The plasma column extends from the surface of the metal electrode to the surface of the water. Figure 11 is a photo of the plasma column.

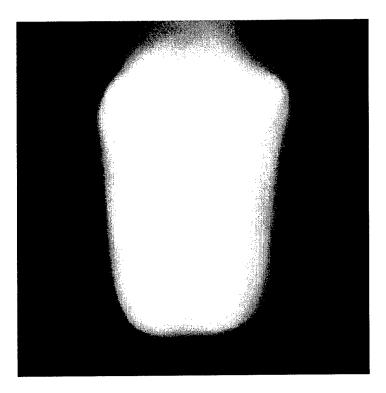


Fig. 11 Appearance of the air discharge. The plasma column is 2 cm long.

Preliminary measurements of the gas temperature using the rotational structure of the second positive system of nitrogen showed that the gas temperature remains relatively low (below 2000 K). Using Optical Emission Spectroscopy, we have also conducted some preliminary studies on the identification of the species generated in such a plasma. Figure 12 shows an example of emission spectra covering the 300-700 nm wavelength range. The spectrum is dominated by OH,  $N_2$ , and  $N_2^+$  transitions. Note that the emission lines above 600 nm are due to second order light because no proper filtering was used in front of the entrance slit of the spectrometer.

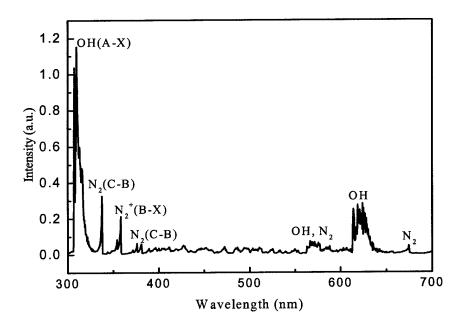


Fig. 12 Emission spectrum from the air plasma discharge

These preliminary results lead us to request new funds to further investigate this novel discharge and its possible applications. More detailed experimental results will be presented in reports pertaining to this new line of research.

## **Collaborations with Other Groups**

The P.I. has been involved in the research activities of the Plasma Rampart MURI Program directed by AFOSR in cooperation with DDR&E. In this context, a good part of the P.I.'s contribution was in developing novel approaches to generate non-equilibrium air plasmas at atmospheric pressure with a relatively low power budget. Collaborations with Prof. K. H. Schoenbach's group at ODU and Prof. Igor Alexeff (Univ. Tennessee) have been ongoing. The P.I. of this project is also a member of the research team at ODU, working on the AFOSR-funded Compact Pulsed Power MURI. Prof. Edl Schamiloglu of the University of New Mexico heads this effort.

Most recently, the P.I. joined the new ODU Center for Bioelectrics (CBE). The CBE is a research center dedicated to the biological/medical applications of cold plasmas and pulsed electric fields. The group led by the P.I. will concentrate a good part of their effort in investigating the interaction of "cold" plasmas with microorganisms. Applications ranging from sterilization to wound healing are contemplated.

#### Other Activities of the P.I.

The P.I. served as a Session Chair at the Pulsed Power Plasma Conference which was held at Las Vegas, NV, June 17-22, 2001. This session was on the Medical,

Biological, and Environmental Applications of Plasmas. The P.I. also organized a session on the same topic at the IEEE International Conference on Plasma Science, which was held in Banff, Canada, May 2002. In addition, the P.I. was a Guest Editor of the IEEE Transactions on Plasma Science Special Issue on the Non-Thermal Medical/ Biological Applications of Ionized Gases, and Electromagnetic Fields. This issue appeared in August 2002.

## List of Papers Supported by this Grant

- M. Laroussi, D. A. Mendis, and M. Rosenberg, "Plasma Interaction with Microbes", *New Journal of Physics*, Vol. 5, pp. 41.1-41.10, 2003.
- M. Laroussi, C. M. Malott, and X. Lu "Generation of an Atmospheric Pressure Non-Equilibrium Diffuse Discharge in Air by Means of a Water Electrode", *In Proc. Int. Power Modulator Conf.*, Hollywood, CA, pp. 556-558, 2002.
- M. Laroussi, X. Lu, and C. M. Malott, "A Non-equilibrium Diffuse Discharge in Atmospheric Pressure Air", *Plasma Sources Sci. Technol.*, Vol. 12, No. 1, pp.53-56, 2003.
- M. Laroussi, J. P. Richardson, and F. C. Dobbs, "Effects of Non-Equilibrium Atmospheric Pressure Plasmas on the Heterotrophic Pathways of Bacteria and on their Cell Morphology", *Appl. Phys. Lett.* Vol. 81, No. 4, pp. 772-774, 2002.
- M. Laroussi, I. Alexeff, J. P. Richardson, and F. F. Dyer "The Resistive Barrier Discharge", *IEEE Trans. Plasma Sci*, Vol. 30, No. 1, pp. 158-159, 2002.
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- J. P. Richardson, F. Dobbs, and M. Laroussi, "Effect of the RBD on Bacterial Viability, Biochemical Pathways, and Morphology", *In Proc. IEEE Int. Conf. Plasma Sci.*, Las Vegas, NV, p. 312, June 2001.
- I Alexeff and M. Laroussi, "The uniform, Steady State, Atmospheric DC Plasma" In Proc. Electromed2001, p. 31, May 2001.
- M. Laroussi, J. P. Richardson, and F. C. Dobbs, "Biochemical Pathways in the Interaction of Non-Equilibrium Plasmas with Bacteria", *In Proc. Electromed2001*, p. 33, May 2001.
- M. Laroussi, J. P. Richardson, and F. C. Dobbs, "Biochemical and Morphological Effects of Non-Equilibrium Atmospheric Pressure Plasmas on Bacteria", Int. Symp. Plasma Chem., Orleans, France, pp. 729 734, July 2001.

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